

BDX-613-2517

Parylene Coating of Syntactic Composites

By A. H. Schloman

Published November 1980

Topical Report
R. J. McWhirter, Project Leader

19960307 008

DEPARTMENT OF DEFENSE
ASTICS TECHNICAL EVALUATION CENTER
ABRADCOM, DOVER, N. J. 07801

Prepared for the United States Department of Energy
Under Contract Number DE-AC04-76-DP00613

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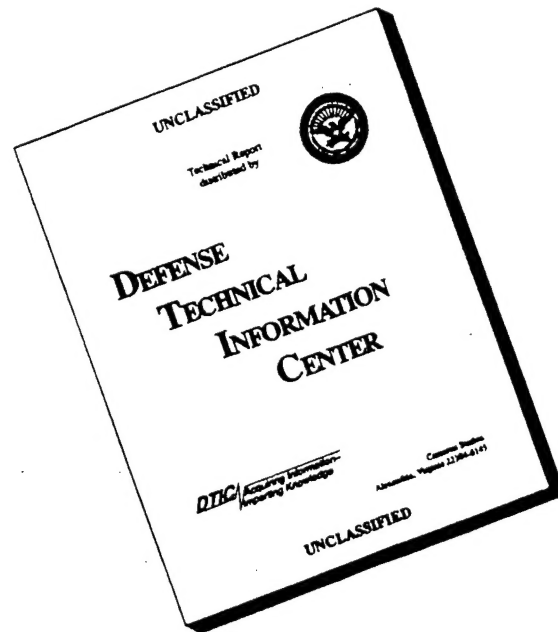
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Printed in the United States of America

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BDX-613-2517
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PARYLENE COATING OF SYNTACTIC COMPOSITES

By A. H. Schloman

Published November 1980

Topical Report
R. J. McWhirter, Project Leader

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**Kansas City
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PARYLENE COATING OF SYNTACTIC COMPOSITES

BDX 613-2517, Topical Report, Published November 1980

Prepared by A. H. Schloman

A manufacturing process developed for parylene coating syntactic parts has resulted in several improvements. Thin edges have been strengthened, which minimizes breakage during the manufacturing process and subsequent assembly; part and surface toughness has been improved; the coefficient of friction during assembly has been reduced; and the bonding of the pads, shoehorn, and clips has been enhanced. Improvements in the tensile strength and flexural strength of the syntactic composite as a result of the coating are discussed, and coated parts and deposited films produced by laboratory and production coaters are compared.

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A prime contractor with the United States
Department of Energy under Contract Number
DE-AC04-76-DP00613

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SUMMARY

A manufacturing process has been developed for parylene coating syntactic foam parts. These parts are machined from syntactic composite billets molded from a blend of polyimide resin, carbon microballoons, and graphite fiber. The final part has extremely thin and fragile edges which can break during manufacturing and subsequent assembly operations. In addition to the need for significantly strengthening these edges, there was also a need for a decreased coefficient of friction, improved bondability with attaching parts, minimized density gradients where stress concentrations could develop, minimized dusting of the surface, and a repeatable process which is independent of an operator. Several different techniques were evaluated in an effort to resolve these problems; however, the parylene process was the only one which met all of the needs.

Initial evaluation of the parylene coating process and its effects on the composite was conducted on a few test samples of miscellaneous size. When the data from these samples indicated that the parylene process met the strengthening requirements, development work was initiated to identify the variables involved in applying the coating to syntactic foam. Since both aft and forward foam parts are machined to their final configuration and then parylene coated, close dimensional tolerances have to be maintained. This necessitated the establishment of a dimer charge weight and a method of obtaining coating uniformity within a part and from part to part. Syntactic foam has a very porous surface which makes it difficult to measure the actual coating thickness; therefore, a coating weight gain was established for each configuration. The coating weight gain is affected by the amount of moisture in the part, which required the establishment of a vacuum drying cycle prior to coating.

Coating data indicate that the parylene coating process increases the flexural strength of the syntactic foam from 3.70 to 6.90 MPa, with an acceptable increase in density. Flexural tests indicated that the parylene also increases the deflection properties of the material, thus allowing the material to flex without failing. The laboratory and production model coaters are capable of maintaining the part coating weight requirements, and the coaters provide good coating uniformity from part to part. DXT data indicated that the coating was uniform within a part. The results from thermogravimetric analysis, tensile tests, and chemical extraction processes indicate that both coaters produce acceptable parylene films.

Vacuum drying tests, using different vacuum levels, were conducted with aft and forward billets containing different amounts of moisture. These tests indicated that a vacuum drying cycle of 2 mm for 2 hours would remove the moisture from the billets.

All the necessary development of the parylene coating process for the syntactic parts has been completed, and no future work is planned.

DISCUSSION

SCOPE AND PURPOSE

Development of a manufacturing process to reinforce the thin edges on syntactic foam spacers was required to reduce breakage of the part during its manufacturing and assembly processes. In addition to the need for significantly strengthening these edges, there was also a need for a decreased coefficient of friction, improved bondability with attaching parts, maintaining a minimal density variation, especially sharp changes in density where stress concentrations could develop, minimized dusting of the surface, and having a repeatable, operator-independent process. The parylene coating process was the only one of the processes evaluated that met all of the requirements.

Very little parylene coating data on composite substrates were available. Therefore, it was necessary to determine the processing effects of the parylene on the syntactic supports. Test samples and parts were coated to evaluate the effects of the parylene on part density, coating uniformity, and tensile and compressive strengths, as well as the effects on flexural strengths. The diameter of the coating chamber of the laboratory coater was not large enough to coat the forward spacers. A larger chamber was fabricated for this purpose, and the data from these coatings were then evaluated.

Included are data on parylene coated parts and films produced by the laboratory and production coaters. These data are evaluated, and the results are compared to determine if the two coaters will give comparable parts and films and will meet the coating requirements.

ACTIVITY

Parylene Coating Process

The parylene coating process, patented by the Union Carbide Corporation, is accomplished by vapor phase deposition. A dimer is used initially rather than a polymer, and in simple compact equipment (Figure 1) the material on the surface of the part is polymerized. To achieve this coating process, the solid dimer (Figure 2) is first subjected to a two-step heating process which converts the dimer into a reactive monomeric vapor. This vapor, when passed over room temperature objects, deposits on all the exposed surfaces, polymerizes, and evenly coats all areas including edges and points. The polymerization process using parylene N dimer is diagrammed in Figure 3.

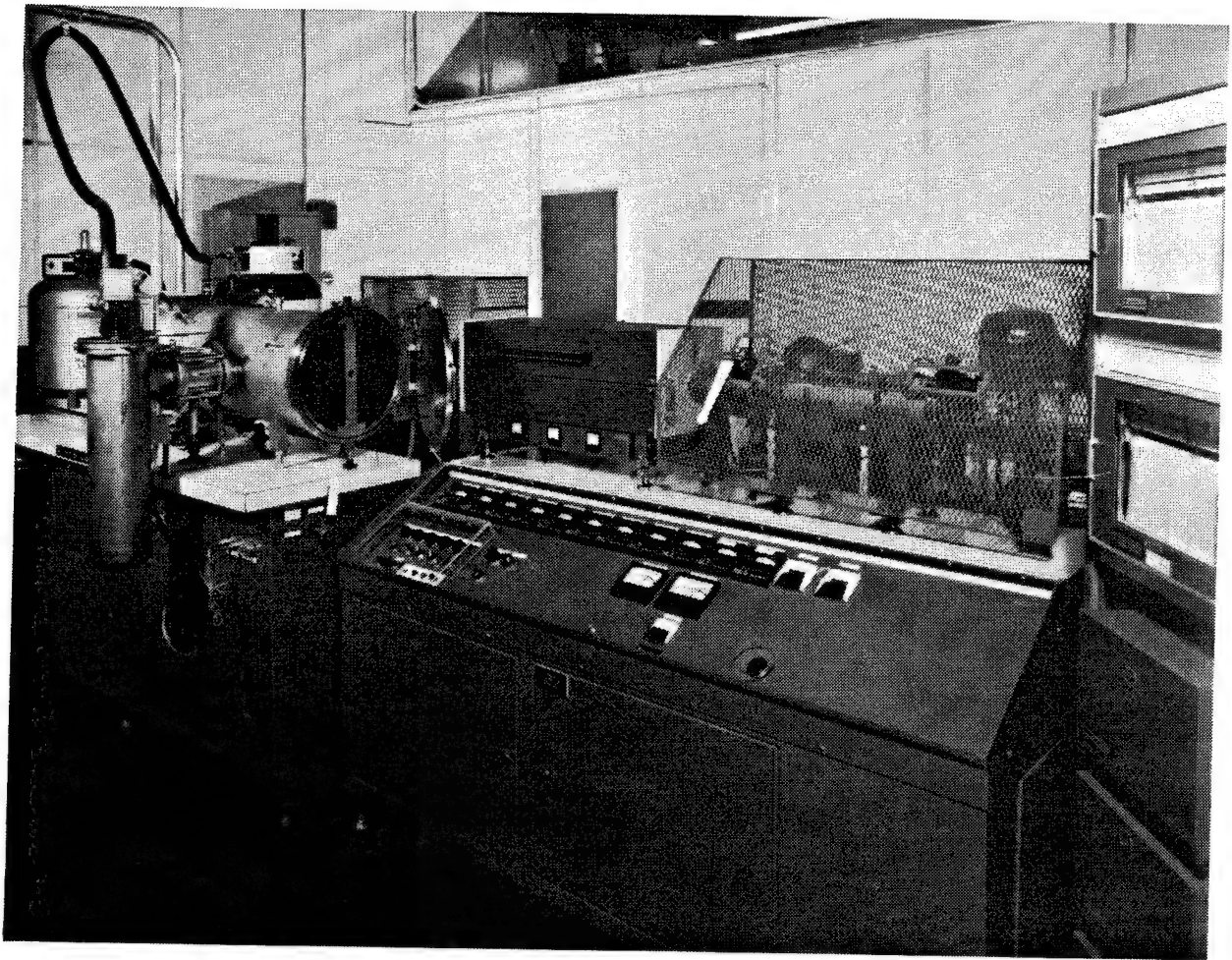


Figure 1. Production Parylene Coater

The parylene coating process (Figure 4) is conducted in an evacuated heated system. The dimer is placed in the vaporizer, and the vapors, when heated, pass through a heated valve and pipes into a tub furnace which heats the material to approximately 680°C and converts it to a monomer. This monomer then passes through heated pipes which direct it into the coating chamber through a header. The header assembly distributes the vapor going into the coating chamber through an adjustable distributor slot (Figure 5). This feature, combined with rotating the parts, helps control the coating uniformity. Monomer that is not deposited on the chamber walls or on the parts is collected by the cold trap.

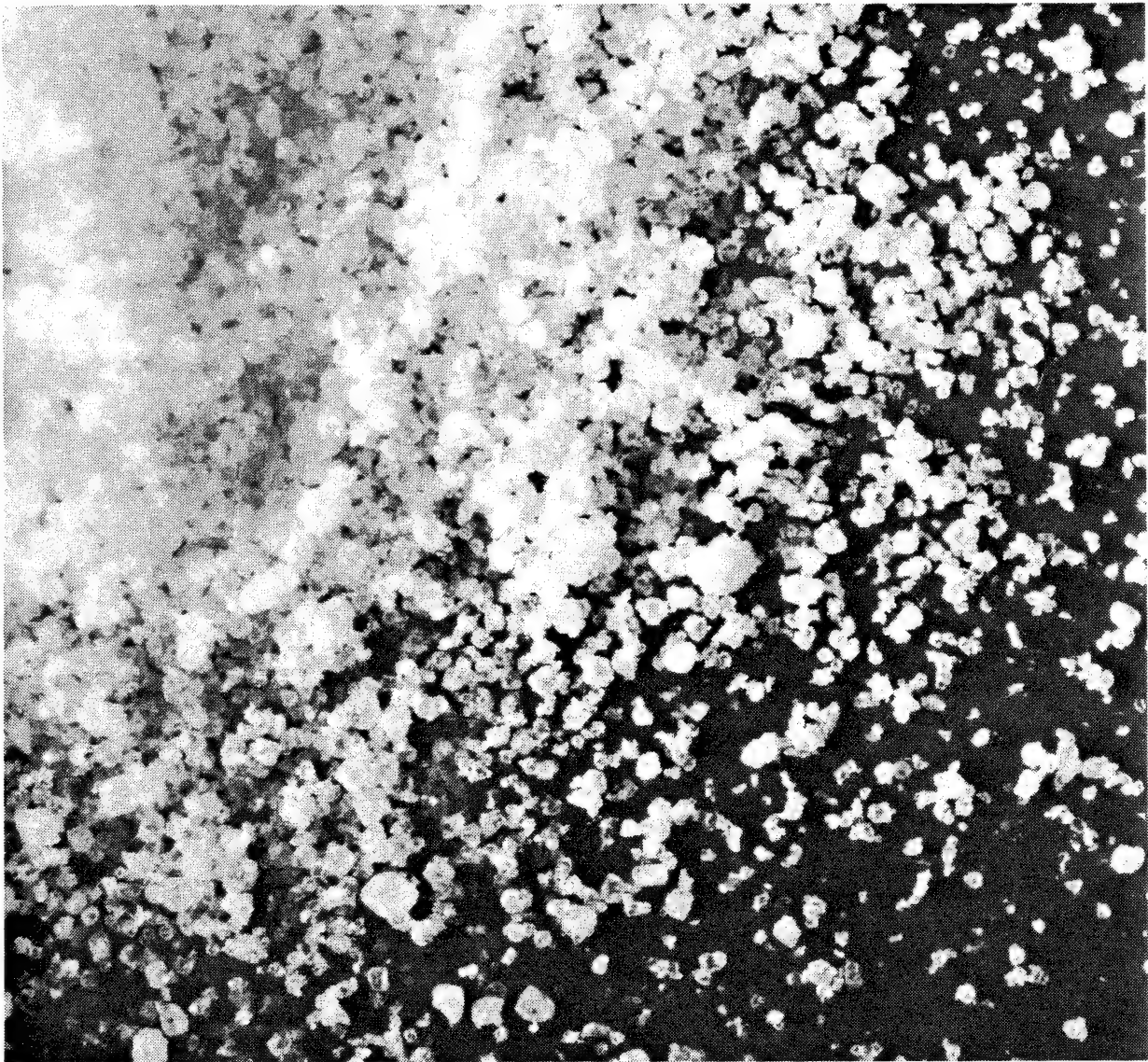


Figure 2. Parylene N Dimer

The parylene coating process is readily controlled and repeatable. The coating thickness is controlled by the amount of dimer placed in the vaporizer. The rate of deposition is controlled by the time required for the dimer to vaporize, which is directly related to the line vacuum level of the system. Therefore, a vacuum controller is used to regulate the heating of the vaporizer which in turn controls the deposition rate. By coordinating the distributor slot opening with a deposition rate, and by using a constant dimer charge weight, uniformity and coating thickness can be

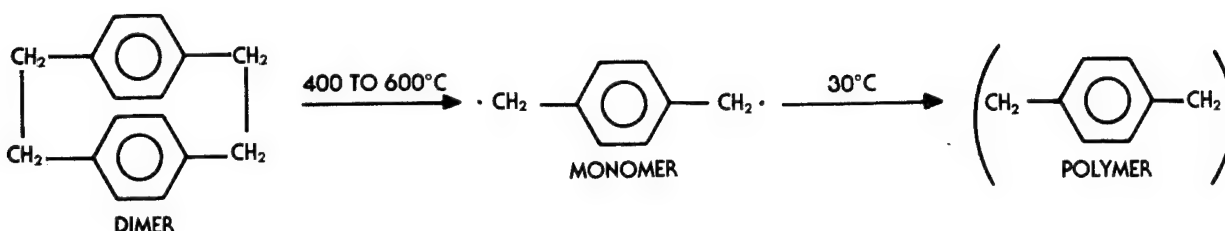


Figure 3. Parylene N Polymerization

controlled very closely and the coating will be very uniform from run to run and from part to part.

The coater temperature and vacuum controls are mounted on the equipment cabinet (Figure 6). When the coater is in the automatic operating mode, the vacuum controller is connected to an alarm to notify the operator of any loss of vacuum, and the temperature controllers will also alarm if the temperature is above or below the set point. These features allow the equipment to be operated with a minimum amount of attention.

The parylene coated syntactic foam part (Figure 7) represents the thin edges of the aft spacer and illustrates the advantages of parylene coating over other strengthening methods. The principal advantage is that the parylene penetrates the syntactic foam in such a manner that the coating density is greater near the outer surface, and its density decreases as the depth of penetration increases. This decrease in density eliminates the abrupt line of reinforcement formed by other strengthening methods, which allow the foam to fail in the area adjacent to the reinforcement. The parylene also encapsulates the individual carbon microballoons as it penetrates the porous foam, thus bonding them together with a thin, flexible film. The film allows the syntactic foam to deflect without breaking. The heavier parylene coating near the surface gives the foam very good handling properties, and it reduces the coefficient of friction between the part and its housing during assembly.

Scanning Electron Microscope (SEM) photographs (Figures 8 and 9) provide a closeup of the parylene coating on the foam surface and the penetration of the parylene into the porous foam. These photographs indicate that the penetration of the parylene into the foam is about 0.4 mm, which is enough to completely penetrate the thin sections of the parts.

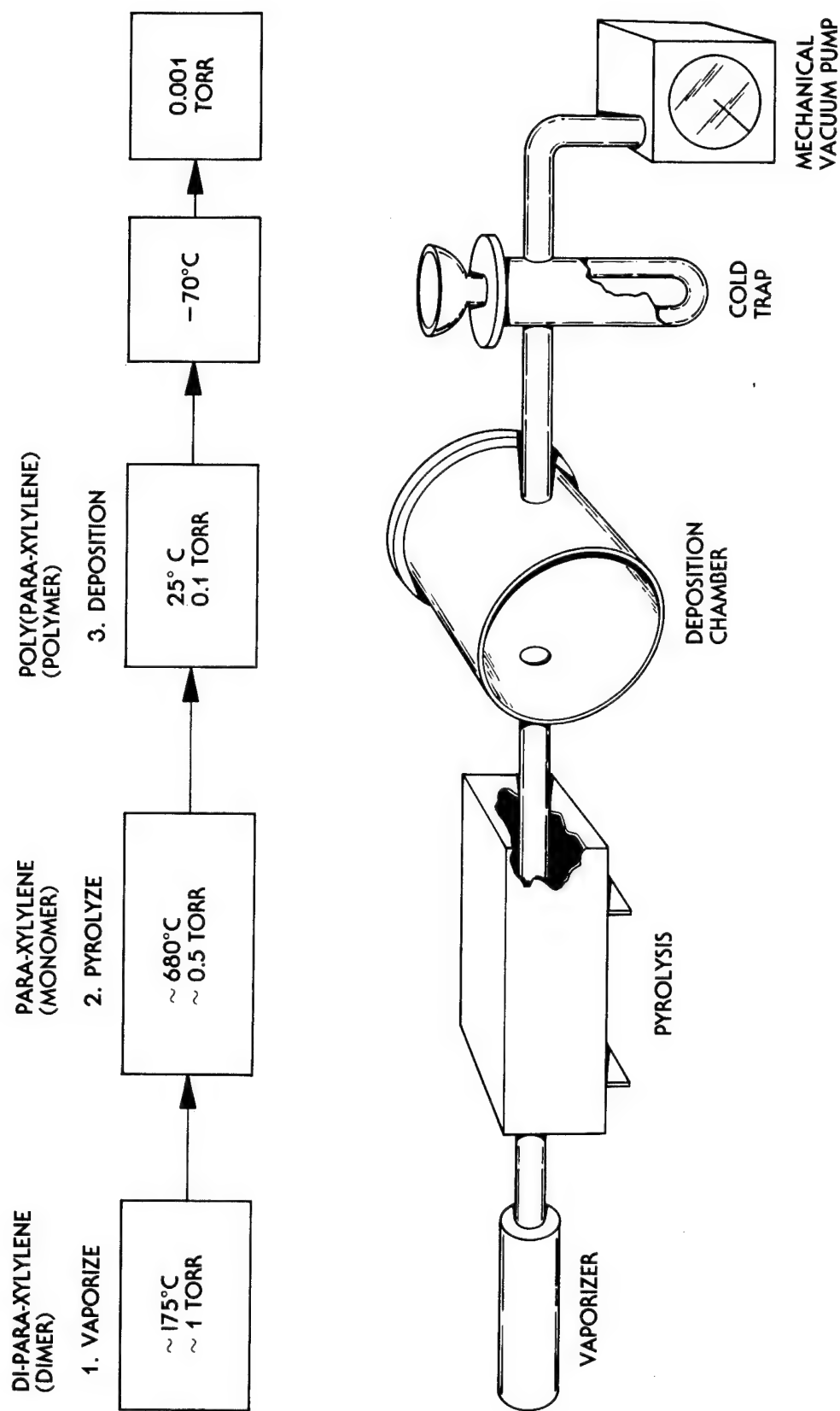


Figure 4. Parylene Process Diagram

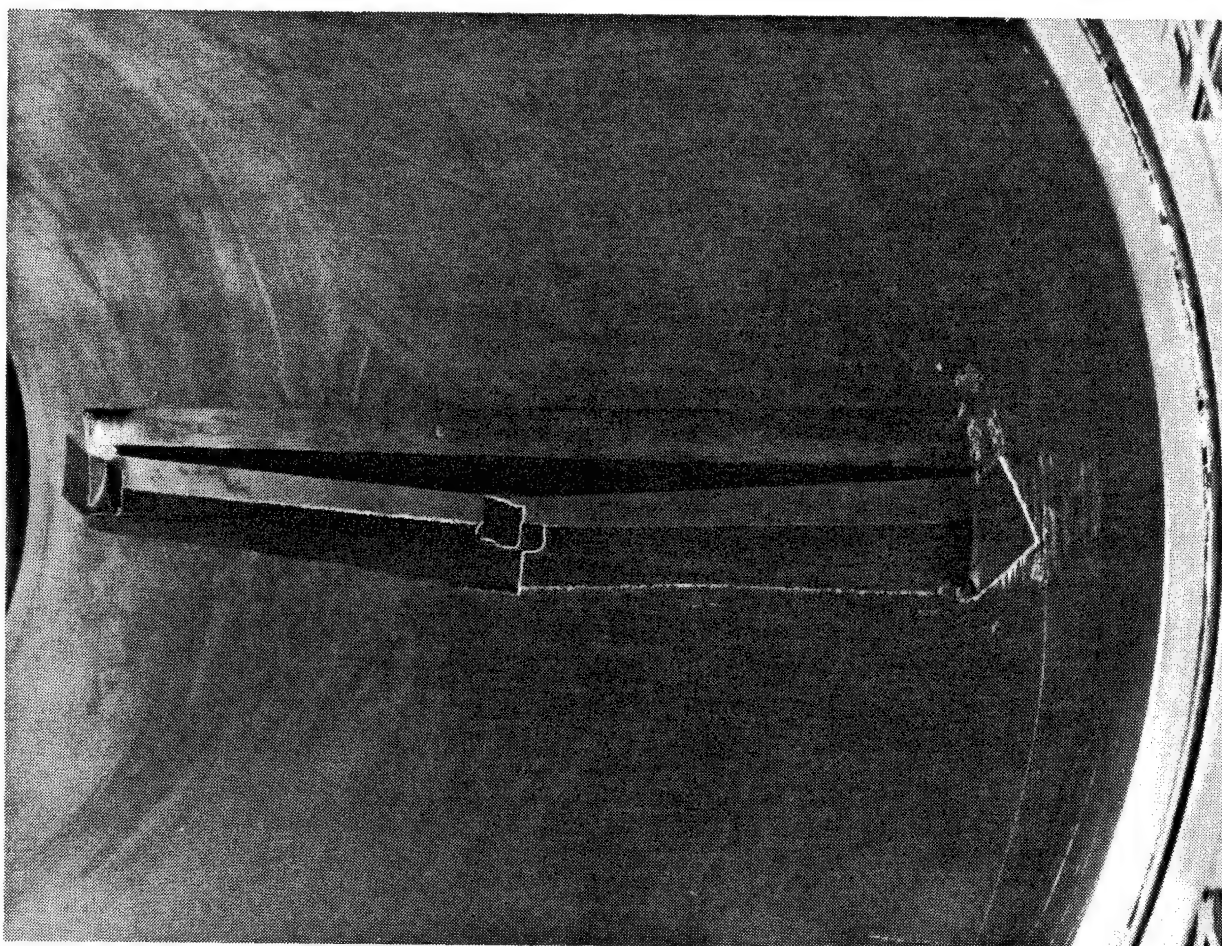


Figure 5. Coating Chamber Distribution Slot

Laboratory Coater Evaluation

The laboratory coater was used to coat test samples and paid order parts while the production model coater was being procured. The laboratory coater originally had a coating chamber 305 mm in diameter by 762 mm long, but the forward spacer required a chamber with a larger diameter. Therefore, an aluminum chamber 340 mm in diameter by 762 mm long was fabricated. The production coater has a coating chamber the same size as that of the laboratory model.

A parylene coating run consists of either two aft spacers or three forward spacers, because these combinations offered the largest amount of coating area that could be placed inside the coating chamber, and the larger the coating area the better the

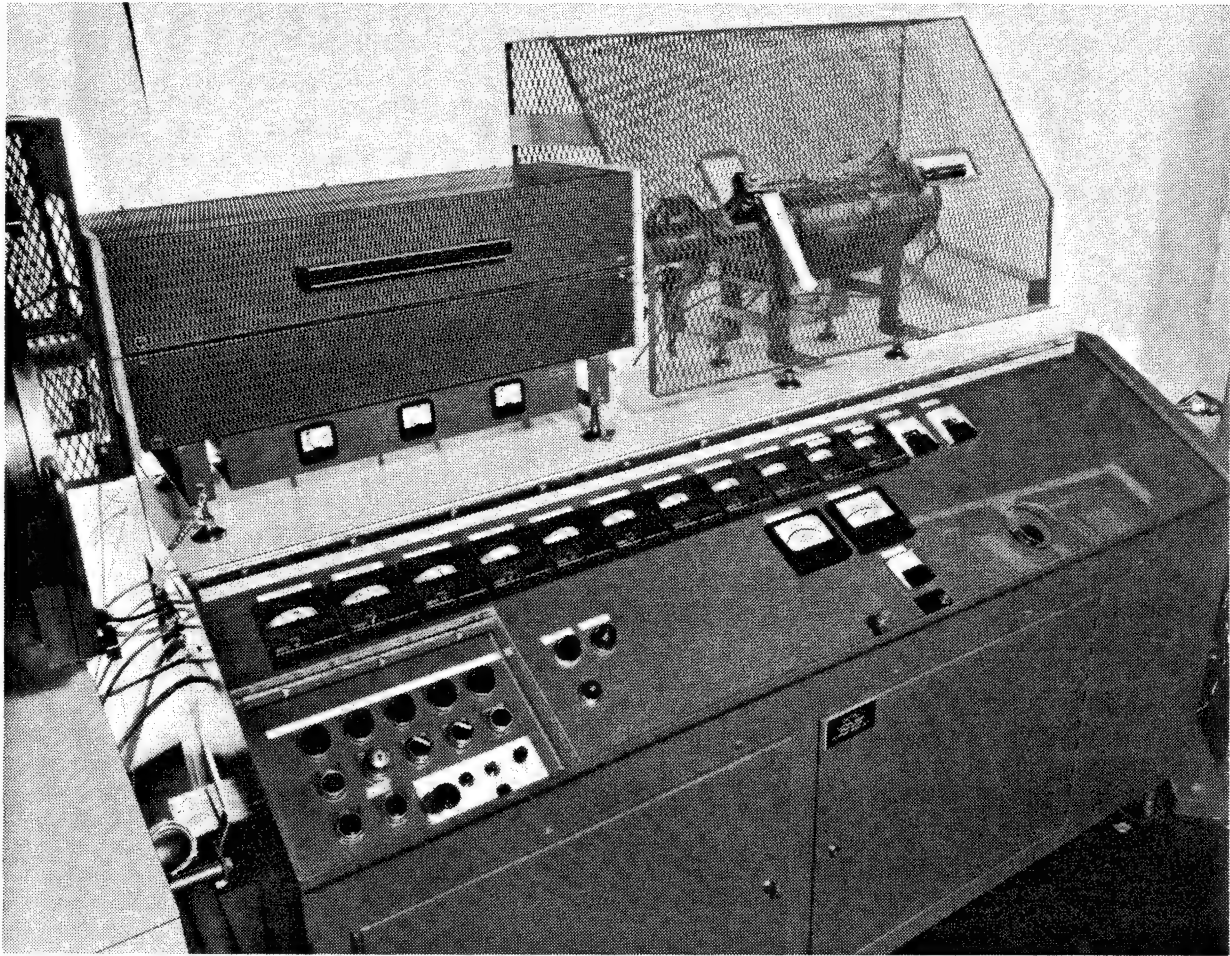


Figure 6. Parylene Coater Control Cabinet

coating efficiency. Coating efficiency, as shown in Tables 1 and 2, is the ratio of total part weight gain to the dimer charge weight. The average coating efficiency was 75 percent for the forward spacers and 58 percent for the aft spacers.

Since the syntactic material is very porous, aluminum witness strips were placed on the coating fixtures and coated with the parts to try to determine a representative coating thickness and to detect coating variations from run to run and within a run. The witness strip thickness data (Tables 1 and 2) indicated that the inner supports had larger thickness variations than did the outer supports. Since the films are very thin, these variations could be the result of measuring errors.

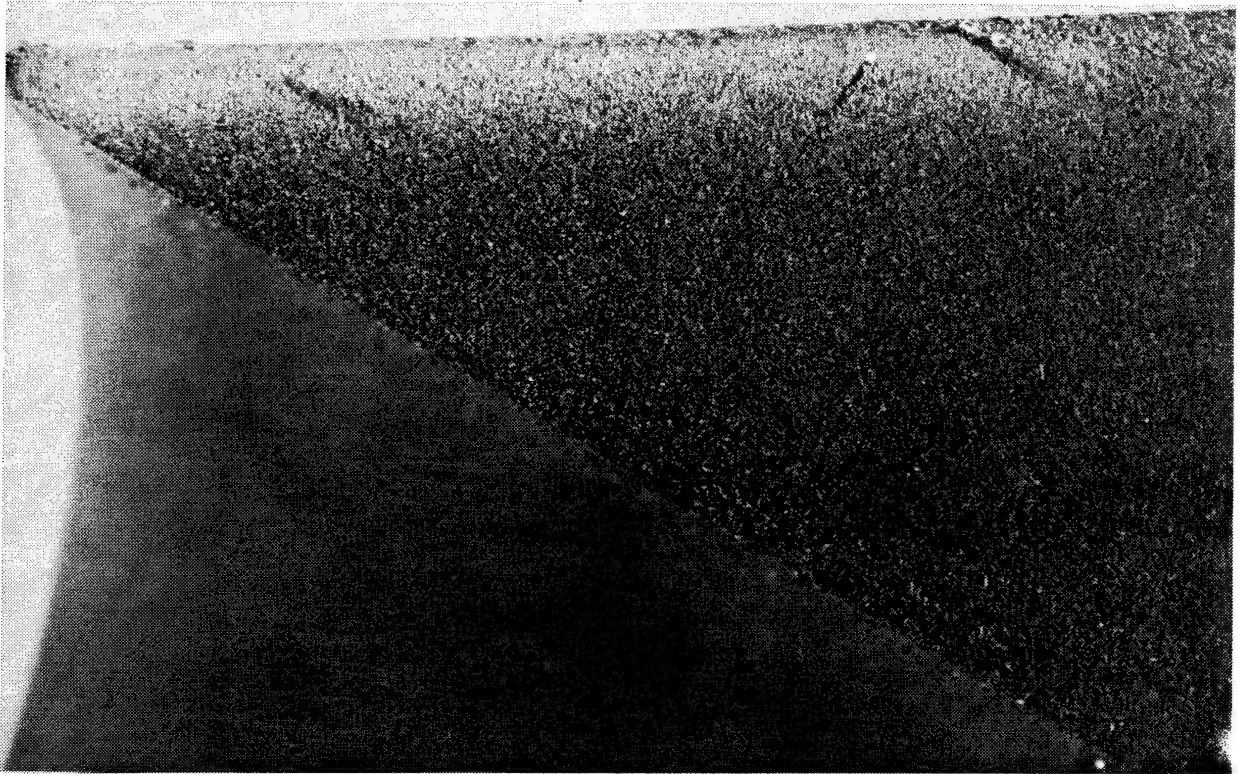


Figure 7. Coated Part Cross Section

The parylene coating is applied after the parts have been machined. This manufacturing process requires the parylene coating to be uniform and repeatable from part to part and within a part. To determine the coating buildup and uniformity, several test cylinders were machined and measured. The parylene was applied; the measurements were retaken; and a second coating was then applied. The coating buildup, or thickness, for both runs is tabulated in Table 3. The coating thickness deviation on the first run was larger than that on the second coating, which was probably because the uncoated foam is very hard to measure, being very porous and very soft. Obtaining an accurate diameter is very difficult; but once the parylene coating is applied, the surface is more durable and accurate measurements can be taken.

Aluminum witness strips were attached to the test cylinders in three places: the window or front of the chamber, the center, and the inlet or rear of the chamber. These witness strips did not have the same coating thickness as the parts, principally because the parts and the witness strips have different surface

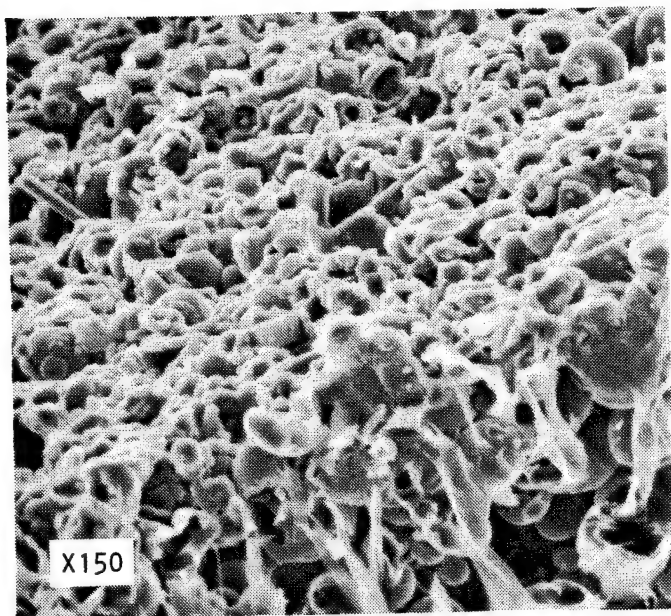


Figure 8. Parylene Coating on Syntactic Foam (SEM Photograph)

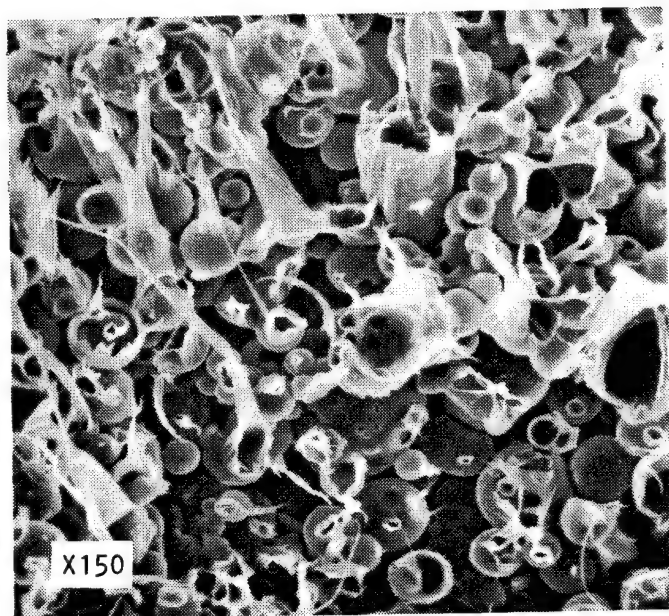


Figure 9. Penetration of Parylene Coating Into Syntactic Foam (SEM Photograph)

Table 1. Summary of Data From Laboratory Coater Run on Aft Spacers

Run Date	Part Serial Number	Average Witness Strip Thickness (μm)	Charge Weight (g)	Part Weight Gain (g)	Total Weight Gained (g)	Coating Efficiency (Percent)
2/8	2/8-1 2/8-2	11.8	150	43.5 46.2	89.7	60
2/22	709 705	7.2	120	36.0 34.5	70.5	59
2/23	710 716	7.0	120	36.0 34.5	70.5	59
2/25	731 738	6.6	120	34.5 36.5	71.0	59
3/13	572* 575*	6.6	120	33.0 33.0	66.0	55
3/22	667 678	9.2	120	31.1 36.0	67.1	56
3/24	730 734	6.6	120	37.0 38.0	75.0	63
3/24	762 763	5.5	120	36.0 34.0	70.0	58
4/5	783 786	6.7	120	37.0 35.0	72.0	60
4/5	807 787	7.6	120	39.0 39.0	78.0	65
4/6	801 844	11.3	120	40.0 38.0	78.0	65
4/6	797** 792**	8.9	120	36.0 36.0	72.0	60
5/9	869 875	8.8	120	36.0 35.0	71.0	59
5/9	873 872	7.4	120	33.0 35.0	68.0	57
5/10	876 893	7.0	120	33.0 34.0	67.0	56
5/10	892	3.6	55	23.0	23.0	42

*Parts were special, grooved cylinders.

**Test blocks were coated along with parts.

Table 2. Summary of Data From Laboratory Coater Run on Forward Spacers

Run Date	Part Serial Number	Average Witness Strip Thickness (μm)	Charge Weight (g)	Part Weight Gain (g)	Total Weight Gained (g)	Coating Efficiency (Percent)
2/8	570 557 543	2.6	120	34.2 28.5 31.0	93.7	78
2/9	571 Dummy 579	2.6	120	31.3 30.0 30.0	91.3	76
3/17	556 566 576	3.9	100	25.0 24.0 24.0	73.0	73
3/22	557 553 540	9.6	100	21.7 25.3 27.6	74.6	75
5/17	721 726 796	2.2	100	26.0 26.0 25.0	77.0	77
5/17	793 802 811	2.6	100	26.0 24.0 24.0	74.0	74
5/18	580 712 806	*	100	24.0 24.0 24.5	72.5	73
*Witness strips ruined						

textures. The witness strips did indicate, however, that on the first coating part numbers 563 and 567, located near the inlet, should and did have a thicker coating. With better measuring surfaces on the second coating run, the coating thickness of the parts more closely approached the witness strip thickness (Table 4).

Witness strips are not attached to the surface of the parts, and the coating could not be removed from the foam surface so that it could be measured. Therefore, two billets were machined

Table 3. Test Cylinder Coating Thickness Data

First Coating Thickness (μm)*				Second Coating Thickness (μm)*			
Test Cylinder				Test Cylinder			
560	563	565	567	560	563	565	567
19.7	78.7	19.7	39.4	39.4	39.4	0	19.7
39.4	39.4	19.7	59.1	59.1	39.4	39.4	19.7
19.7	39.4	39.4	59.1	59.1	39.5	19.7	19.7
0	177.2	39.4	19.7	39.4	59.1	0	19.7
19.7	19.7	0	39.4	0	39.4	0	19.7
78.7	0	59.1	19.7	0	19.7	59.1	19.7
19.7	0	0	19.7	19.7	0	0	0
19.7		98.4	39.4	19.7	59.1	19.7	39.4
		0	39.4	0	0	39.4	0
		19.7		59.1	0	19.7	
				19.7			
				19.7			
Average							
27.08	50.63	29.54	37.21	27.91	29.55	19.70	17.51
Standard Deviation							
23.37	62.11	31.12	15.40	22.94	23.22	20.77	11.84
*Readings taken at random locations							

to conical shapes with constant wall thickness, and DXT traces were run before and after coating. Four longitudinal DXT traces 90 degrees apart were run on each part, and the traces were compared to determine the coating uniformity. Two representative traces are reproduced in Figure 10, and the changes in density along the part and within the part are tabulated in Table 5. The DXT data show that the parylene coating is very uniform in each trace position and within each part.

Table 4. Test Cylinder Witness Strip Data

Strip Location	Coating Thickness (μm)			
	Test Cylinder			
	560 and 563		565 and 567	
	First Coating	Second Coating	First Coating	Second Coating
Window	21.7	19.7	7.9	21.7
	17.7	19.7	7.9	37.4
	17.7	19.7	7.9	19.7
	19.7	23.6	7.9	29.5
				21.7
Center				19.7
	15.7	15.7	Witness Strip	15.7
	21.7	15.7	Ruined	17.7
	15.7	13.8		15.7
	17.7	15.7		17.7
Inlet	19.7	15.7		
	31.5	19.7	11.8	17.7
	33.5	19.7	11.8	19.7
	25.6	19.7	11.8	17.7
	39.4	19.7	11.8	17.7
Average	33.5		11.8	17.7
	23.6	18.5	10.2	20.5
Standard Deviation	7.9	2.8	2.0	5.9

Production Coater Evaluation

The production model parylene coater was purchased from Union Carbide Corporation, Bound Brook, New Jersey. The acceptance coating requirements stated the coating uniformity to be within ± 10 percent, as measured on witness strips attached to aluminum coating fixtures, with an average film thickness of 19.5 μm . Four coating runs, two on each configuration, were conducted. The results are listed in Table 6. The coater met the requirements and was shipped to Bendix for installation.

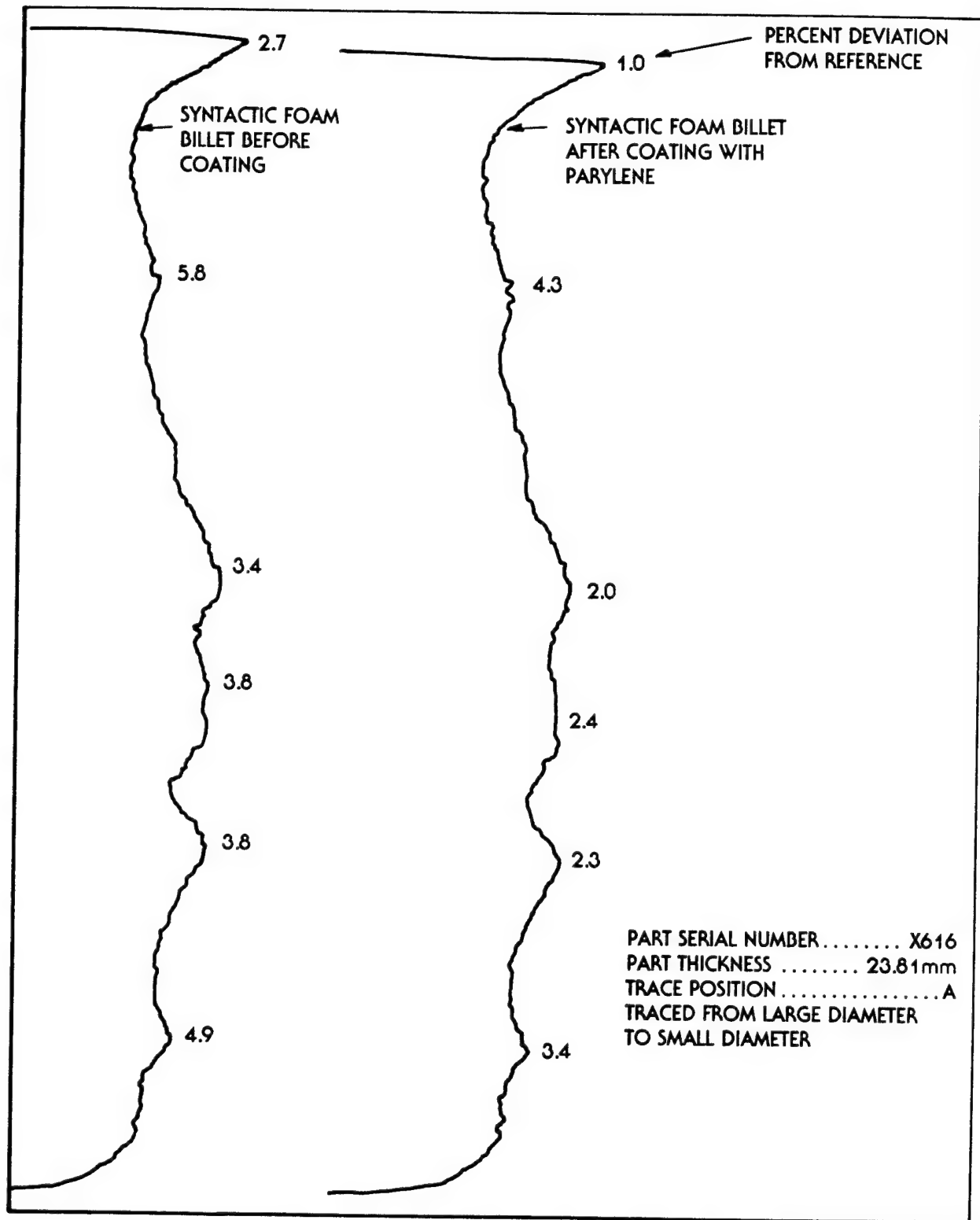


Figure 10. DXT Evaluation of Coating Uniformity

Table 5. DXT Data on Coated and Uncoated Test Billets

Trace Position	Test Billet	
	616	561
	Percent Change	Percent Change
A	1.5	8.4
	1.5	7.4
	1.4	6.8
	1.4	6.2
	1.5	6.8
	1.7	
B	1.3	
	1.3	4.9
	1.6	4.9
	1.9	4.5
	2.0	4.8
	2.6	
C	2.5	
	2.3	4.5
	2.0	4.6
	1.8	4.8
	2.0	4.9
	2.9	
D	1.4	
	1.6	5.9
	1.2	6.1
	1.2	6.6
	1.3	5.8
	1.2	
	1.5	
	1.7	

After the coater was installed, an equipment engineer from Union Carbide Corporation supervised the initial startup and checkout. The equipment operated satisfactorily during the checkout runs and was available to coat the syntactic supports for PPI.

Table 6. Summary of Production Coater Acceptance Data

Run Number	8	9	10	11
Substrates	3 Forward	2 Aft	3 Forward	2 Aft
Dimer Charge Weight (g)	82	69	75	69
Coating Rate (g/min)	0.42	0.32	0.39	0.29
Number of Data Points	179	72	180	80
Average Thickness (μm)	22.4	21.3	2.05	1.99
Standard Deviation (μm)	1.40	0.95	1.37	1.09
Percent Standard Deviation	6.4	4.5	6.7	5.5

The aft spacers with the aluminum witness strips are mounted on the coating fixture. The fixture masks the inside surface so that they will not be coated, but allows a vacuum to be drawn on the inside of these parts. Preliminary evaluation with the laboratory coater indicated that the system pumpdown time can be reduced if the inside is evacuated instead of removing the trapped air by pulling it through the walls of the part.

Coating data on the syntactic parts revealed that the production coater has an average coating efficiency of about 76 percent on the aft spacers and a coating efficiency of about 86 percent of the forward spacers. The part coating weight was increased to 35 \pm 5 grams for the aft and 30 \pm 5 grams for the forward spacers. Examination of Tables 7 and 8 indicates that after a charge weight adjustment, all the parts meet the weight gain requirements.

Tables 9 and 10 list the witness strip coating measurements taken with a bench micrometer anvil force set at 170 grams. These values indicate that the coating uniformity throughout the chamber is very good and that from run to run the average coating thickness does not change by more than three microns.

Table 7. Summary of Data From Production Coater Run on Aft Spacers

Run	Part Serial Number	Average Witness Strip Thickness (μ m)	Charge Weight (g)	Part Weight Gain (g)	Total Weight Gained (g)	Coating Efficiency (Percent)
1	896 895	10.2	100	38.0 38.0	76.0	76
2	899 901	21.2	90	33.5 34.0	67.5	75
3	903 902	21.6	90	32.0 34.0	66.0	73
4	904 905	21.0	90	35.0 33.0	68.0	76
5*	2001 2002	17.2	90	34.0 36.0	70.0	78
6*	2003 2004	15.2	90	35.0 33.0	68.0	76
7*	2005 2006	14.6	90	33.0 36.0	69.0	77

*PPI Parts

To determine the effects of parylene on compressive, tensile, and flexural strengths, a group of compressive and tensile test specimens 28.68 mm in diameter by 25.4 mm high, and some flexural test samples 12.75 by 50.80 by 2.54 mm thick were machined from blocks 203.20 by 203.20 by 38.10 mm thick. The data from these samples indicate that the flexural strength is increased while the modulus is decreased; but the greatest advantage of the parylene is that it allows the foam to bend before it fails. Tensile test samples were prepared from 0.30-g/cm³ foam with different surface-to-volume ratios, and during the coating the specimens picked up different amounts of parylene which gave parts with different densities. As with the flexural strength, the parylene coating improved the tensile strength. Table 11 lists the tensile, flexural, and compressive strength data.

Table 8. Summary of Data From Production Coater Run on Forward Spacers

Run	Part Serial Number	Average Witness Strip Thickness (μm)	Charge Weight (g)	Part Weight Gain (g)	Total Weight Gained (g)	Coating Efficiency (Percent)
1	840 859 863	15.7	120	30.0 38.0 37.0	105.0	88
2	854 867 870	16.1	110	28.0 31.0 32.0	91.0	83
3*	1001 1002 1003	11.1	110	31.0 33.0 31.0	95.0	86
4*	1008 1009 1010	18.8	110	30.5 33.0 33.0	96.0	88
5*	1004 1005 1006	14.6	110	32.0 32.0 30.0	94.0	85
6*	Dummy 1011 1007	17.0	110	29.0 35.0 33.0	97.0	88
7*	925 928 930	17.8	110	33.0 33.0 31.0	97.0	88

*PPI Parts

Comparison of Laboratory and Production Coaters

The laboratory and production model coaters produced comparable data in the areas of coating weight requirements and TGA analysis of the parylene films. The laboratory and production coaters are capable of maintaining the coating weight requirements for both of the syntactic parts. After the correct charge weight had been established, the coaters produced acceptable parts in all the coating runs.

Table 9. Witness Strip Coating Thickness for PPI
Aft Spacers

Strip Location	Run		
	3	6	7
	Thickness (μm)		
Door	17	12	15
	16	12	14
	17	11	16
	17	10	15
	16	13	14
	17	14	16
Center	17	13	15
	16	16	13
	17	15	15
	17	12	14
	18	14	19
	16	16	17
Drive	19	20	14
	17	21	13
	17	20	12
	19	19	12
	18	18	15
	20	20	16
	17	15	15
	1	4	2
Average Thickness	16	15	14
Standard Deviation	5	4	3

The production model coater had better witness strip thickness uniformity and coating efficiency. The difference in film thicknesses could be affected by several factors. The witness strip films from the laboratory coater were measured by using a dial indicator and comparator stand, but the production coater films were measured with bench micrometer. Also, the laboratory coater witness strips were mold released; but the films used for the coater acceptance runs and evaluation runs were not mold released.

Table 10. Witness Strip Coating Thickness for PPI Forward Spacers

Strip Location	Run			
	1	2	4	5
	Thickness (μm)			
Door	8	14	12	16
	11	11	13	18
	9	15	13	2
	9	16	12	17
	10	14	14	17
	9	16	11	19
Center	13	21	19	18
	11	22	14	16
	14	23	13	17
	14	24	14	16
	10	20	16	16
	12		20	19
Drive	1	19	16	15
	13	21	18	16
	14	20	15	17
	11	19	14	20
	10	21	15	17
	9	22	16	15
	11	19	15	17
	2	4	2	2
Average Thickness	11	18	14	16
Standard Deviation	3	5	4	4

The mold release may not have been applied uniformly which could have caused some of the thickness variations.

The two major differences between the coaters is that the production model coater requires less parylene material or dimer to provide the required coating weight gain of each configuration, and the production coater requires a minimum of operator attention.

Table 11. Properties of Standard and Parylene-Coated Foam

Property	Parylene-Coated Foam			Standard Foam	
	Test Value	Density (g/cm ³)		Test Value	Tested Density (g/cm ³)
		Original	Coated		
Compressive Strength (MPa)					
RT	4.06	0.297	0.306	5.86	0.299
200°C	3.79	0.297	0.306	5.52	0.299
Compressive Modulus (MPa)					
RT	268	0.297	0.306	---	0.299
200°C	321	0.297	0.306	---	0.299
Flexural Properties at RT					
Strength (MPa)	5.62	0.30	0.40	3.83	0.292
Modulus (MPa)	507	0.30	0.40	556	0.292
Deflection (m/m)	0.0015	0.30	0.40	0.0007	0.292
Tensile Strength at RT (MPa) for Three Coating Densities					
	2.17	0.30	0.315	1.24	0.267
	2.05	0.30	0.336	2.00	0.300
	3.28	0.30	0.359	2.21	0.333

Vacuum Drying

Syntactic foam will absorb moisture, and this moisture must be removed before the parts are coated so that accurate coating weights can be obtained. If the moisture is not removed before the parts are weighed and placed inside the coater, the coater vacuum will remove this moisture and the part weight after coating will be in error. The initial vacuum drying work was done by exposing aft and forward billets to a vacuum of 94.5 kPa. Some billets which were placed in desiccated containers lost very

little weight. After placing the billets in a vacuum for 7 hours they lost most of the moisture. After an additional 9 hours of drying, the billets lost only a few more grams of moisture but never obtained the reference weight. The reference weight is the weight of the billet immediately after postcure and should be moisture free.

Additional aft billets were subjected to high relative humidity and gained about 8 grams of moisture. These parts were placed in a 400-Pa vacuum for 1 hour and 3 hours. The weight of the parts returned to within 1 gram of their reference weight within 1 hour, and after 3 hours in the vacuum no additional weight was lost.

These drying cycle tests were conducted on billets because it should be more difficult to remove moisture from the molded billet than it would be from porous surfaces of a machined part.

Material Properties

Evaluation of the parylene films produced by both coaters was conducted to determine if the coaters produced films of comparable quality. The evaluation consisted of thermogravimetric analysis (TGA), tensile tests of films, and dimer content analysis by thermal evaporation.¹ Film samples from the coating chamber and cold trap residue were evaluated by using a mass spectrometer.

Thermogravimetric analysis of parylene films for five runs, three from the laboratory coater and two from the production coater, were conducted in atmospheres of both air and nitrogen. The coater runs were made at fast and slow deposition rates, and dry ice was used for the coolant in the cold trap for the first run. All other runs were made with the use of liquid nitrogen as the coolant. The runs were made with the same dimer lot and dimer charge weights.

The curves from the TGA analysis indicate that the films produced by both coaters are nearly the same. The decomposition temperatures for the films in a nitrogen atmosphere range from 464 to 470°C (Table 12). Some of the temperature variation may be caused by the way the tangent lines to the curves are positioned on the curves. Figure 11 provides a representative view of these curves.

The TGA analysis of the parylene films in an air atmosphere indicated that a reaction occurs at about 210°C, and the parylene gains some weight; then at approximately 255°C decomposition starts, with a final decomposition occurring at about 480°C. Table 13 lists the data obtained from the TGA curves, and Figure 12 is a typical TGA curve in an air atmosphere.

Table 12. Thermogravimetric Analysis of Parylene Films,
Using Nitrogen Atmosphere

Laboratory Coater Run	Deposition Rate (g/min)	Percent Weight Loss	Decomposition Temperature (°C)
1	0.91	0.3	467
2	1.38	0	470
3	0.40	0.5	464
Production Coater Run			
8	0.94	0.6	464
9	0.49	1.2	466

The dimer content in the parylene films were analyzed by thermal evaporation and by mass spectrometry. The thermal evaporation method was recommended by Union Carbide Corporation,¹ and the results of this analysis (Table 13) indicate that the production coater has a higher dimer content in its film than does the laboratory coater. This is confirmed by the weight loss in the TGA runs. This amount of dimer is not high enough to affect the film quality. The negative dimer content numbers obtained from the laboratory coater films could be result of weight measurement error.

Mass spectrometric analysis of the films from the coating chamber door and residue from the cold trap indicated that the maximum amount of outgassing occurs over the range of 40 to 85°C. The outgassing products are predominantly monomer C_8H_8 and lesser amounts of dimer $C_{16}H_{16}$ and trimer $C_{24}H_{24}$. As the temperature is raised above 85°C the trimer increases in intensity while the dimer and the monomer become about equal in intensity, with the monomer the most abundant.

The outgassing products from the cold trap residue samples include the monomer, dimer, and trimer with an abundant amount of saturated hydrocarbon. This hydrocarbon material is not aromatic in structure, has a low molecular weight, and has almost all outgassed when the temperature reaches 65 to 70°C. The hydrocarbon is probably the cause of the small pressure spike encountered during

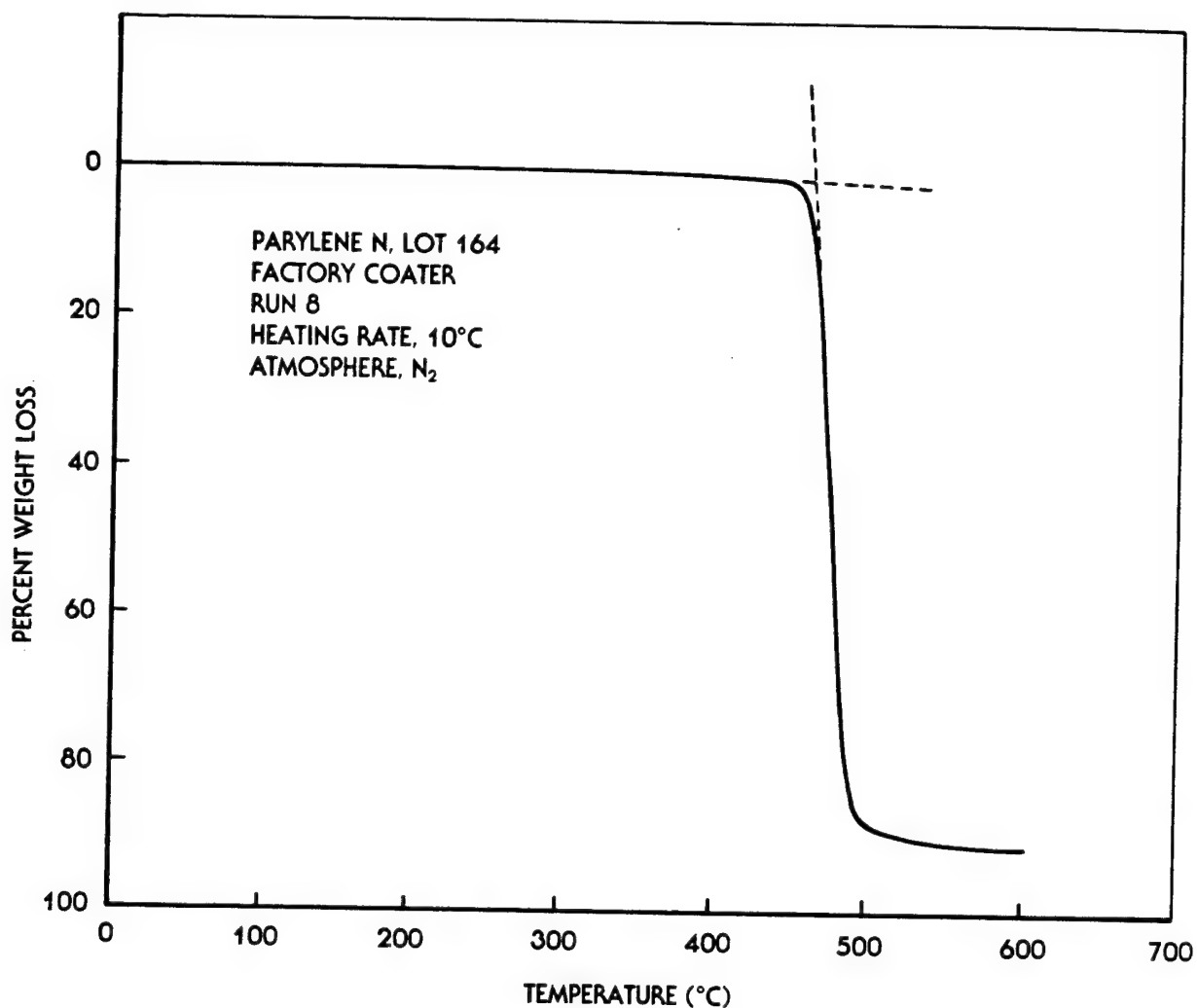


Figure 11. Thermogravimetric Analysis of Parylene Film in a Nitrogen Atmosphere

some of the coating runs. Like the door film samples, the most abundant outgassing of the cold trap samples occurs over a temperature range of 40 to 90°C.

To evaluate the film tensile strength of both coaters, films were deposited on ferrotype plates and strips 12.7 mm wide by 89 mm long were cut out with a razor blade and straight edge. These films were attached across a window, 38 mm wide by 51 mm long, cut out of a card 76 mm by 130 mm, and the ends were taped down with pressure sensitive tape (Figure 13). The card was placed in the grips of the tensile test machine and aligned, and then the two sides of the card were cut. At this point, the specimen was ready to be pulled.

Table 13. Thermogravimetric Analysis of Parylene Films, Using Air Atmosphere

Laboratory Coater Run	Dimer Content (Percent)	Temperature for Weight Gain (°C)	Start of Decomposition (°C)	Final Decomposition (°C)
1	-0.31	210	250	472
2	-0.15	205	255	477
3	0	215	255	486
 Production Coater Run				
8	0.33	210	262	485
9	0.78	208	263	492

Note: Dimer content determined by vacuum drying

The data obtained from the tensile tests indicated that both coaters produce films of nearly equal strength. The average film strength obtained by the laboratory coater and production coater was 36.17 and 36.81 MPa, respectively. The modulus of the films from both coaters was almost the same. Table 14 provides a summary of the tensile data from 30 specimens.

ACCOMPLISHMENTS

Development of the parylene coating process resulted in the following benefits. The parylene increased the flexural strength to a value that allows the parts to be manufactured and assembled with minimal breakage, and data indicate that the coating is uniform throughout the part. A vacuum drying process was established, along with the dimer charge weights, for coating both configurations; and both coaters are capable of maintaining and controlling the weight gain of each part. After the dimer charge weight had been established, all parts met the weight gain requirements. The production coater has an automatic deposition rate control which makes the actual coating operation independent of an operator, and the entire coating process requires a minimum amount of an operator's attention. Finally, the information and data gathered in using the laboratory coater aided in reducing

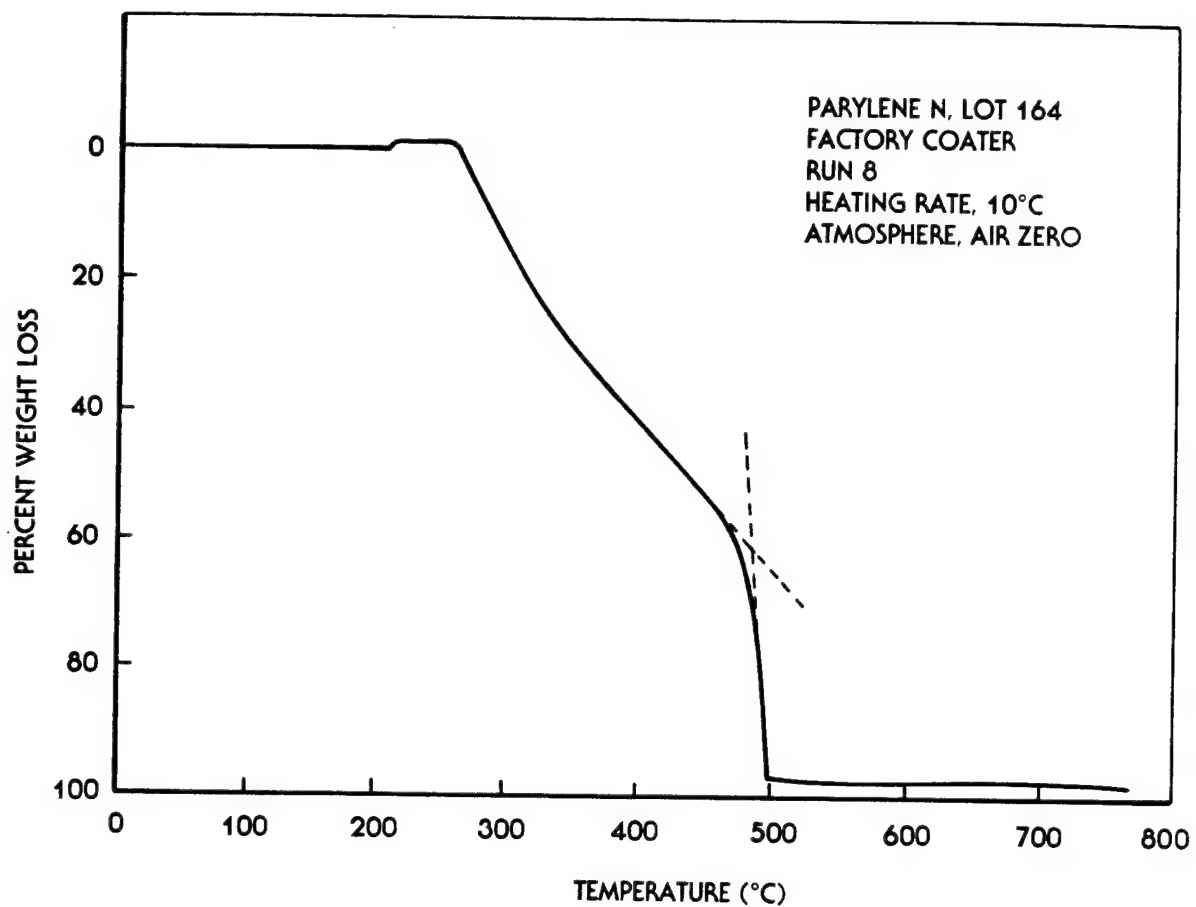


Figure 12. Thermogravimetric Analysis of Parylene Film in an Air Atmosphere

the evaluation and prove-in time of the production parylene coater.

FUTURE WORK

Development of the parylene coating process for the syntactic parts has been completed, and no future work is planned at this time.

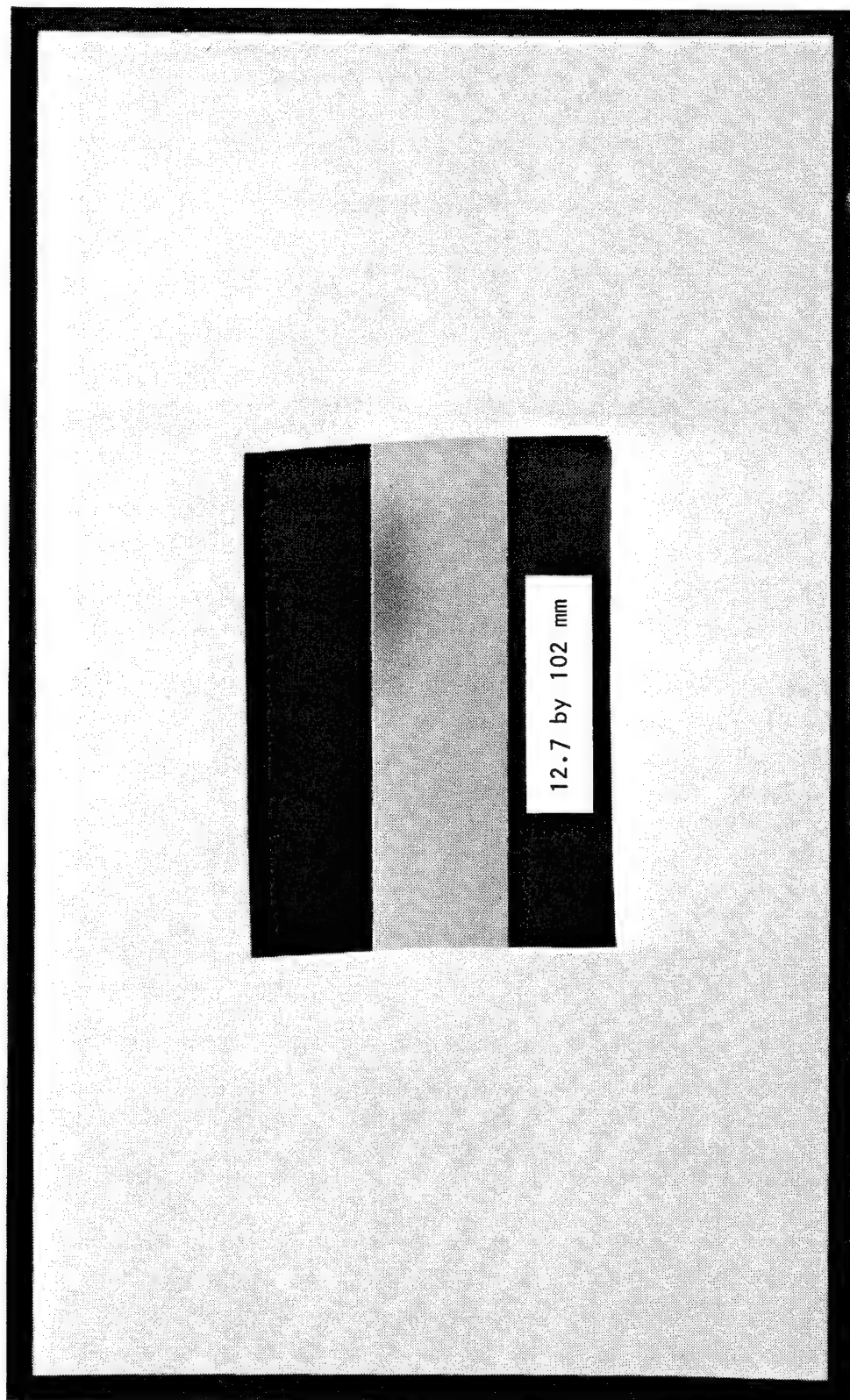


Figure 13. Tensile Test Specimen

Table 14. Parylene Film Tensile Data

Laboratory Coater Run	Thickness* (μm)	Thickness By IR** (μm)	Ultimate Tensile Strength (MPa)	Modulus (GPa)	Elongation (Percent)
1	7.8	6.3	29.9	1.35	4.04
2	14.6	15.4	37.8	1.92	4.24
3	9.1	10.2	40.9	1.47	8.86
Production Coater Run					
8	13.0	11.8	40.9	1.95	4.90
9	11.8	11.0	32.7	1.13	13.72
*Measured with Dial Comparitor					
**Infrared Spectroscopy					

REFERENCE

¹The Determination of Dimer Content in Parylene Films. Bound Brook, New Jersey: Union Carbide Corporation, Standard Testing Method Number WC-160-K, November 2, 1970.

BDX-613-2517

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A. H. Schloman, Topical, November 1980

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PLASTICS: Parylene Coatings

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